Libby - Background Study

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Environmental Services Assistance Team
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Controlling Matrix Interference Effects of Rock Flour In the Fluidized Bed Method For Analysis of Asbestos in Soil

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Abstract

Characterizing Libby Amphibole fibers released from soil to air has historically been performed at the Libby, Montana Asbestos Site by performing Activity-Based Sampling (ABS). The Fluidized Bed Asbestos Segregator (FBAS) method is being investigated as a means to simulate ABS activities in a controlled laboratory environment. However, glacial rock flour found in much of Libby caused many FBAS samples to overload quickly when the method was applied to background soil samples. FBAS analysis on background soils was put on hold by EPA until ESAT determined if the rock flour problem could be mitigated. ESAT determined that much, but not all, of the rock flour could be separated from the analyte of interest (Libby Amphibole asbestos) by deliberately overloading the FBAS filter and allowing the coarsest rock flour to settle out from aqueous suspension for several hours during indirect sample preparation. There are not enough data points in this report to determine a detection limit for LA in rock flour-rich soil by this technique but it may be a considerable improvement over the traditional method and may be < 0.005% (expressed as weight percent LA in soil). A visual examination of a plot of LA soil concentration versus FBAS results indicates a rough linear correlation for the data points in this study. However, ESAT recommends that a statistical treatment be applied to the data by a third party to determine how well the data correlates. ESAT recommends that TEM-FBA analysis of background soils resume by the indirect prep technique described in this report after the SAP is finalized.

Acronyms and Definitions

ABS	Activity-Based Sampling
Aerodynamic diameter	The diameter of a water droplet that would have the same terminal velocity when falling through still air as the particle of interest
BK	Background
ESAT	Environmental Services Assistance Team
equant	Said of a particle with approximately equal dimensions in all directions
FBAS	Fluidized Bed Asbestos Segregator
LA	Libby Amphibole asbestos
ISO	International Organization for Standardization
MCE	Mixed Cellulose Ester
ml	milliliter, a unit of volume, equal to one-thousandth of a liter
µm	micron, a unit of length, equal to one-millionth of a meter
ND	Not Detected
PE	Performance Evaluation
SPF	Sample Preparation Facility
TDF	Technical Direction Form
TEM	Transmission Electron Microscopy
TEM-FBA	The valid value name in the database for processing soils by FBAS and analyzing the resulting filters by TEM, referring to the entire method from start to finish
EPA	United States Environmental Protection Agency
USGS	United States Geological Survey

1.0 Introduction

The Fluidized Bed Asbestos Segregator is a device for measuring the concentration of releasable asbestos fibers in soil. The method was developed by EPA Region 10 in cooperation with the Idaho National Laboratory and is now being applied to the Libby Site. However, recent attempts to apply the FBAS method to background soils were complicated by the rock flour found in many of the BK soils. Much of the Libby Valley was at one time a glacial lakebed, and the glaciers ground down rocks to an extremely fine powder called rock flour, which settled out of the water column in the ancient glacial lake. This rock flour is now found in many of the undisturbed (background) soils in the town of Libby. When the FBAS method is applied to BK soils rich in this rock flour, the filters overload quickly unless a small soil size (typically 0.5 to 0.75 grams) is used. A small soil sample size in the FBAS results in a higher analytical sensitivity and sub-sampling of a smaller, and possibly less representative, fraction of the original soil sample.

Conversely, if a larger sub-sample of the original soil sample is used in the FBAS, the results may be more representative of the original soil sample as a whole, and analytical sensitivity will be improved. This will only be practicable if a method can be found to keep the rock flour from overloading the filters quickly, while still capturing the amphibole asbestos fibers of interest. Separation of the rock flour from amphibole asbestos is a difficult problem because they are chemically and mechanically similar.

Photo 1. The FBAS at the EPA Region 10 Lab in Port Orchard, Washington. All photos in this paper were taken by ESAT Region 8.

The success or failure of pre- or post-treatment techniques to mitigate the effects of rock flour can be quantitatively evaluated by seeing how fiber density of the filters analyzed in the TEM produced by those techniques compare to control filters prepared in the normal manner for a given soil concentration. It is also important to see if the new technique(s) result in an improvement to analytical sensitivity. Fiber density is important because it directly relates how much can be learned about a sample for a given amount of analytical effort or cost. The more that the fibers can be separated from the rock flour, the higher the fiber density will be for a given particulate loading and soil concentration. Analytical sensitivity can always be improved by analyzing more grid openings, but this correspondingly increases analytical cost.

From April 20 to 22, 2011, three ESAT Region 8 staff members visited Jed Januch at the EPA Region 10 Laboratory in Port Orchard, Washington to investigate possibilities to improve the FBAS method as it applies to soil samples rich in rock flour. Additional FBAS runs for the Rock Flour Study were performed at ESAT's Sample Preparation Facility in Troy, Montana from June 1 to 3, 2011.

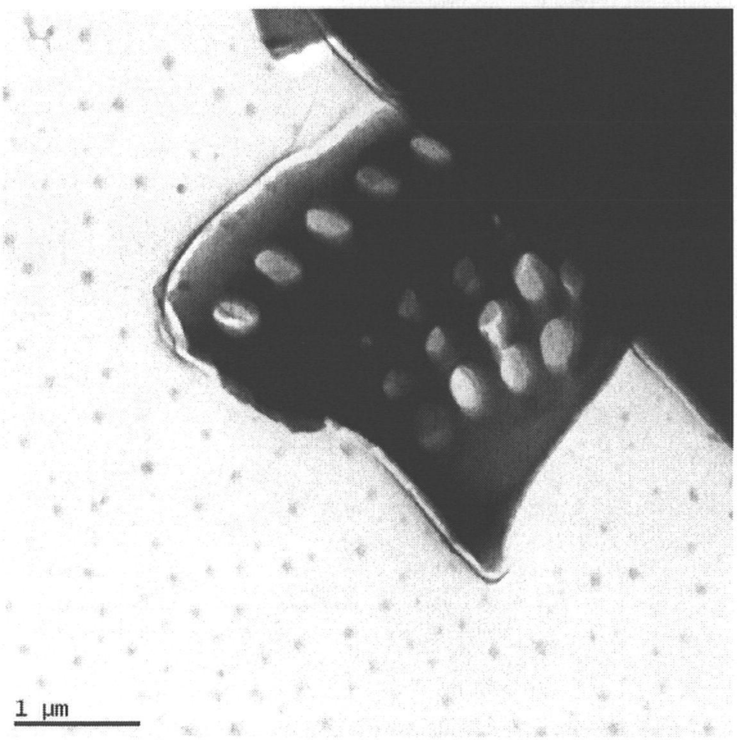

Photo 2: Diatoms are commonly seen in FBAS samples that have rock flour and may have lived in the ancient glacial lake at Libby. This one was found in a BK soil.

2.0 Development of Internal Reference Materials for the Rock Flour Study

ESAT set out to prepare soils of known LA concentration that were as realistic as possible in how well their composition and grain size distribution compared to those of actual Libby BK soils. So, for the matrix of the spiked soils, ESAT used actual Libby soil known to contain a high amount of rock flour. At the Troy, Montana SPF, ESAT Region 8 examined the remaining sample archive of the BK soils that had been processed by FBAS and analyzed by TEM-ISO in the winter of 2010/2011. The FBA fractions (material that passed through a number 20 sieve, with openings 0.85 mm across) of the two heaviest rock flour-rich BK soils from the 1" to 6" depth that had been previously analyzed as ND by TEM-FBA were selected and sent to the ESAT Region 8 Lab in Golden, Colorado. The samples selected were BK-00044 and BK-00076, both of which are low in organics and high in rock flour content and considered by ESAT to be representative of the BK soils by visual examination. The silt and clay size fraction (< 75 μm) as determined by EMSL/New Jersey by sieving of the (unprocessed) BK-00044 and BK-00076 soil samples was 40% and 53% by weight respectively, which is typical of the range found in the 40 BK soils for which petrographic analysis was performed. At the lab in Golden, ESAT mixed together the sieved (FBA) fractions of these two soils and spiked the mixture with an old PE soil, which consists of USGS's Arvada soil that had been spiked with 1.6% by weight Libby Amphibole asbestos. In this manner, ESAT produced rock flour-rich sieved BK soil with the following concentrations (expressed as weight percent) of Libby Amphibole: 0.1%, 0.05%, 0.01%, 0.005%, 0.001%, and 0.0003%. The contribution of the Arvada soil to the internal reference materials is minimal and does not constitute more than 10% of the any one reference sample's weight. Because these spiked soils were not prepared and characterized by an outside third party, they are referred to as internal reference materials and not as PE's. ESAT packed these spiked soils into a cooler with various other supplies, and shipped them to the Region 10 Lab ahead of the trip.

3.0 Control Samples and Analytical Methods

To serve as a control, three replicates of each reference concentration (expressed as percent Libby Amphibole by weight) of rock flour-rich internal reference materials were ran through the FBAS under completely normal conditions (0.5 grams of soil ran through the FBAS for 3 minutes with no pre- or post-treating of the sample material). The starting weight of 0.5 grams was selected because based on ESAT's experience it would produce a properly-loaded filter for soils of this composition. The resulting filter cassettes for the 0.1% LA soil FBAS runs were shipped to the ESAT Region 8 Lab in Golden, Colorado. The filter cassettes for the FBAS runs on the other 5 soil concentrations were sent to the EMSL/Libby Lab. At the analytical labs, the control filters were all prepared directly and analyzed by TEM according to the ISO 10312 method and all applicable standard Libby Lab modification forms.

TEM examination at the Region 8 Lab of the direct preparations of the FBAS filter runs on the 0.1% LA rock flour-rich spiked soil revealed that most of the rock flour consists of angular quartz, feldspar, and mica grains from 1 to 7 microns across, although a few grains were larger and many were smaller. A small amount of clay mineral grains are also present but they do not contribute significantly to the rock flour. The quartz and feldspar rock flour grains are of a more or less equant shape; the mica grains are in the shape of small flakes. Unfortunately, quartz, feldspar, and mica are all silicate minerals just like amphiboles are. They are all highly durable just like asbestos is. For this reason, ESAT did not make any attempt during the Rock Flour Study to separate fibrous amphiboles from the rock flour by chemical methods. Only mechanical methods were investigated to see if they could (at least partially) separate the two. All of the FBAS filter cassette samples from the non-control samples were prepared and analyzed at the ESAT Region 8 Lab in Golden, Colorado. The analyses were performed by TEM according to the ISO 10312 method and all applicable standard Libby Lab modification forms.

For the purposes of the Rock Flour Study, analyses of all samples followed the same filter loading rule that was followed for the 2010 Background Study. This rule is now followed for all FBAS samples analyzed in support of the Libby Project. Specifically, this rule requires that all filter preparations for FBAS samples that are actually examined in the TEM (whether prepared directly or indirectly) have to be from 10% to 30% loaded by area, without obvious uneven loading. If a filter is 25% to 30% loaded, the TEM analyst has the option to not analyze the sample based on professional judgment if too many overlapping particles are observed and request a new filter submittal. If the filter is <10% or >30% loaded by area, a new filter submittal must be obtained. The purpose of this rule is that:

1) Since FBAS is a soil-to-air asbestos method, the only item of interest is characterizing fine silicate particulates. Analytical time spent on anything else does not characterize the asbestos content of the soil. If a sample is too lightly loaded, analytical effort is spent unnecessarily to examine "empty" areas of the filter. If that is the case, it is then preferable to re-run the soil through the FBAS at a higher starting sample weight to load the filter more heavily to capture more particles of interest and also to lower analytical sensitivity.
2) Conversely, if a sample is too heavily loaded, overlapping of particles will obscure asbestos, even to the point of covering asbestos fibers or creating arrangements of particles where both ends of an asbestos fiber are obscured by non-asbestos debris. Such fibers cannot be counted, and results are then biased low.

4.0 Soil Pre-Treatment by Suspension and Filtration

Various pre-treatments of the spiked rock flour-rich soils were attempted before they were mixed with clean quartz sand and poured into the FBAS. None of the pre-treatment methods investigated by ESAT were found to be successful. Only post-treatment as described in section 7 below was found to be successful in mitigating the rock flour problem. The first pre-treatment attempted was to weigh out 5 grams of the 0.1% LA rock flour-rich internal reference material, suspend and mix it in a 100 ml aqueous solution, and allow the solution to settle for 15 minutes in hopes that most of the rock flour would settle out and leave most of the asbestos still suspended. The aqueous suspension was mixed and poured into a graduated cylinder and after 15 minutes the top half (50 ml) was pipetted off and filtered through a 37 mm 0.45 micron pore size polycarbonate filter. However, the filter clogged quickly so it took a very long time (approximately 20 minutes) to filter the 50 ml of suspension through the filter.

Of the original 5 grams of rock-flour rich soil in the aqueous suspension, only 0.187 grams of the soil was recovered from the top half of the suspension from the graduated cylinder after settling for 15 minutes. This 0.187 grams of soil was the fine fraction that was captured on the filter, which was dried, collected from the filter, disaggregated, and then ran by FBAS in the usual manner. However, after TEM analysis, no improvement in LA fiber density was found in the resulting FBAS filter relative to the controls.

The same approach was attempted with 2-propanol to serve as the solvent in the solution. In this run, 5 grams of the 0.05% rock flour-rich soil was mixed in 100 ml of 2-propanol and allowed to settle in a graduated cylinder. After 15 minutes, the top half of the suspension was drawn off and filtered through a 37 mm 0.45 micron pore size polycarbonate filter. The filtration went much faster with the alcohol than with water because the capillary nature of water impedes filtration. However, only 0.088 grams of soil were recovered by the filtration, which was then mixed with clean quartz sand and processed by FBAS. No improvement in LA fiber density was found in the resulting FBAS filter relative to the controls.

Based on the TEM analytical results, ESAT concluded that pre-treatment of the soil by suspension and settling of the soil in water or alcohol before running it by FBAS is not a viable option for mitigating the rock flour problem. Potential limitations of pre-treating the soil by aqueous suspension are:

1) A high analytical sensitivity if the weight of soil recovered by filtration is small.
2) The flakes of dried mud recovered from the filter may not completely break up, so the fibers within them may not be released during subsequent FBAS processing.

5.0 Soil Pre-Treatment by Mechanical Vibration

Next, pre-treatment of soil by mechanical vibration was attempted. The objective was to cause the coarsest particles to settle to the bottom and the finest particles to rise to the top. Three replicates of 3 grams each of the dry 0.1% LA rock flour-rich soil were poured into porcelain crucibles. The crucibles were then sonicated for 3 minutes. Upon visual examination, it was observed that the sonication action had, as expected, preferentially vibrated the finest particles to the top of the soil in the crucibles. The top 0.5 grams of soil was collected from each of the 3 crucibles and ran through the FBAS in the usual manner. This approach was apparently not adequate in separating out the rock flour, because no improvement in LA fiber density was found in the resulting FBAS filters relative to the controls.

6.0 Cyclone Filtration

Region 10 has made a modification to the FBAS so that FBAS samples can be processed onto cyclone filters. The cyclone is a small plastic or aluminum device placed directly in front of the air sampling filter cassette; they are routinely used for respirable dust sampling in occupational settings such as coal mines. Early indications on samples processed by Region 10 show that this method may produce more even loading on the FBAS filters, resulting in fewer fibers being obscured by particulate clumping on the filters. The FBAS cyclone filtration method involves a higher Q_R (of 0.125, corresponding to a flow into the filter of 2 liters per minute) than the traditional method, resulting in a correspondingly improved analytical sensitivity. Q_R is the ratio of flow through the filter cassette to the total flow in the FBAS system and has the value of 0.0125 in the traditional method. Air is drawn from the glass vessel of the FBAS into the cyclone. Inside the cyclone, the air is spun rapidly, and heavier airborne particles fall to the outer edge of the cyclone and settle into a trap at the base of the cyclone. The cyclone is designed such that the 50% cutoff point is at 4 μm aerodynamic diameter, meaning that 50% of 4 μm aerodynamic diameter particles will pass through the cyclone to the air sampling filter.

The cyclone uses 37 mm diameter, 0.8 μm pore size MCE filter cassettes. The traditional FBAS method uses 25 mm diameter, 0.8 μm pore size MCE filter cassettes. ESAT purchased one cyclone for the Troy SPF. Four samples, at 0.5, 1.0, 2.0, and 5.0 grams of soil, of the 0.05% LA rock flour-rich internal reference material were processed by FBAS at the Troy SPF onto filters with the cyclone. However, none of the resulting filters were properly loaded. ESAT analyzed the most heavily-loaded cyclone filter anyway and no LA fibers were found in the analysis and the filter was only 2% loaded. ESAT asked Jed Januch of EPA Region 10 if he could provide any guidance. Mr. Januch explained that ESAT should check all of the connections in the back of the FBAS to see if we had missed anything when converting the FBAS from normal operation to cyclone operation or if there had been an impediment to the airflow. Just one improper or loose fitting in the connections could have made the difference between success and failure. This was ESAT's first attempt at the cyclone method and additional effort will be needed by ESAT to learn to perform the method successfully. ESAT does have a concern that the cyclones are not that easy to decontaminate between sample runs, and sample throughput was one of the design considerations of the FBAS method.

7.0 Indirect Preparation with Aqueous Suspension and Settling

A considerable improvement was achieved in LA fiber loading relative to the controls when filters were deliberately overloaded by the FBAS and then prepared by indirect-ashed where the aqueous suspension was allowed to settle for several hours. Analytical sensitivity was also generally improved by this technique. The coarsest rock flour grains settled out, leaving Libby Amphibole fibers in suspension so that they could be captured by filtration onto a secondary filter. The inspiration for this method was the Elutriator Method, which was developed by Dr. James Webber of the New York State Department of Health, and Stokes' Law. Stokes' Law is an equation that describes the settling velocity of a particle in a fluid to the viscosity and density of the fluid, the acceleration of gravity, and the density and radius of the particle of interest (assuming that the particle has a spherical shape). According to Stokes' Law, the settling velocity of a spherical particle is proportional to the particle's cross-sectional area (all other things being equal). Asbestos as a rule does have a large surface area to weight ratio so hopefully it would settle more slowly than the rock flour grains, which are approximately equant in shape. The objective of the elutriator method is to fractionate out only the respirable-size fraction (e.g., for a dosing agent for toxicity studies) from a large population of fibers (e.g., from ground-up rock-form asbestos). The elutriator accomplishes this by allowing fibers to settle down through a column of water that flows upward very slowly. Based on the Elutriator paper (see references) and personal communication between ESAT and Dr. Webber, ESAT learned that asbestos fibers settle out of an aqueous suspension based primarily on their diameter, not their length. So, for example, a 0.5 μm diameter asbestos particle with an aspect ratio of 3:1 should sink at about the same rate as a 0.5 μm diameter asbestos particle with an aspect ratio of 20:1.

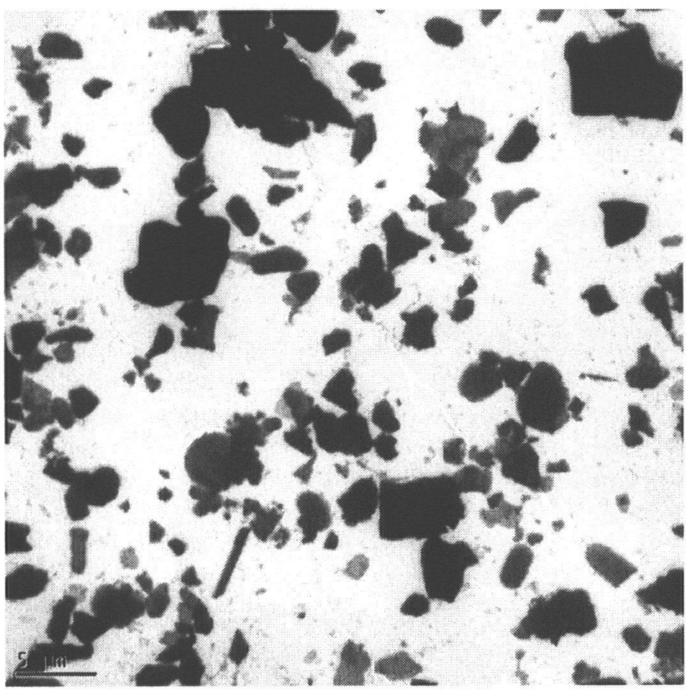

Photo 3: During indirect-ashed preparation, allowing the aqueous suspension to settle for 3 hours removed most of the rock flours grain > 3 μm across. This is a TEM prep of one of the spiked rock flour soils that was ran through the FBAS at 5 grams and prepared in this manner, with a dilution factor (F-factor) of 0.2. The fiber at lower left is Libby Amphibole. The fiber at right is non-asbestos. The scale bar is 5 μm.

So, after a series of trials, ESAT developed the following procedure to run samples by TEM-FBA to optimize the method for analysis of LA in soils rich in rock flour:

1) Run the soil through the fluidized bed at a starting soil weight of 4 or 5 grams (mixed with 16 or 15 grams of clean sieved quartz sand so that the total weight is 20 grams) with the intent of deliberately and heavily overloading the filter.
2) Prepare the filter cassette by indirect-ashed in the normal manner as described in the EPA-Libby-08 SOP. The plasma etcher will burn off all of the organic material so that essentially only silicate materials remain. However, follow these modifications to the standard indirect-ashed procedure:
    a. Following plasma ashing, prepare an aqueous suspension of the sample material with a volume of 100 ml, in a container such as a bottle beaker with a tightly sealed lid. It is the responsibility of the lab to ensure that all particulate debris that was in the original MCE filter cassette (loose inside the cassette, adhering to the cowl, or on the primary filter) is ashed and placed into the aqueous suspension.
    b. Shake the bottle beaker and repeatedly turn it upside-down then upright again to thoroughly homogenize the suspension.
    c. Sonicate the bottle beaker for 3 minutes.
    d. Pour the entire contents of the bottle beaker into a 100 ml graduated cylinder. Record the volume of the suspension to the nearest 1 ml.
    e. Allow the aqueous suspension to settle for 3 hours in the graduated cylinder. Most of the rock flour grains > 3 μm across will settle to the bottom of the graduated cylinder, leaving only fine particles (and asbestos) in suspension.
    f. After the settling time of 3 hours has elapsed, with a 60 ml syringe, pipet off the top 50 ml of the approximately 100 ml in the graduated cylinder. While drawing off the top 50 ml of the suspension, try to not draw any liquid below approximately the 50 ml mark on the graduated cylinder into the syringe. The intent is to only capture the top half of the suspension since the coarsest particles will be in the bottom half. Empty the contents of the syringe into a fresh bottle beaker. Mix the contents of that bottle beaker.
    g. Discard the remaining liquid in the graduated cylinder as ACM water waste in accord with the laboratory's Chemical Hygiene Plan or equivalent.
    h. Prepare a secondary filter by filtering a known aliquot of the top half of the suspension. Save the remaining top half of the suspension in its bottle beaker.
3) The F-factor (dilution factor) is then calculated as the volume of the aliquot that was applied to the secondary filter divided by the volume of the suspension that settled in the graduated cylinder.
4) Prepare the secondary filter in the usual manner and examine it in the TEM. If the sample is from 10% to 30% loaded without obviously uneven loading, it is suitable for analysis. If this is not achieved, prepare a new secondary filter from the top half of the suspension that was saved in step (h) above. Discard all remaining water as ACM waste when the analysis is complete.
5) If none of the secondary filters are properly loaded and all of the top half of the liquid suspension is used up, contact the FBAS laboratory to request a new filter submittal.

One advantage of indirect preparation is that preparing a properly-loaded filter becomes the responsibility of the TEM analytical lab and not the FBAS facility. The TEM labs are highly skilled in producing properly-loaded filters based on their experience. There is also no need for the FBAS facility to estimate what starting weight of a soil will produce a properly-loaded filter, since the resulting FBAS filter will be overloaded no matter what.

Based on ESAT's experience, most, but not all, of the rock flour grains > 3 μm across will be gone after 3 hours of settling. The analyte of interest (Libby Amphibole asbestos) will be correspondingly enriched relative to the rock flour in the remaining fine fraction captured in the top half of the aqueous suspension after 3 hours of settling. The settling time of 3 hours is based on ESAT's experience. Too many coarse particles were observed in the 1 and even 2 hour settling time filters. No appreciable difference could be observed between the 3 and 4 hour settling times. However, 4 hours of settling is difficult from a work-flow standpoint. With plasma etching occurring in the morning and aqueous suspension/settling occurring after lunch, it makes for a long day to filter the secondary filters after 4 hours of settling.

ESAT did have a concern that some PCME fibers would be lost after aqueous suspension. Most of the PCME fibers are narrow (less than about 1 micron in diameter), so they are apparently not falling down through the water column as fast as the medium- to coarse-size rock flour grains are. A review of the data indicates that 51% of all direct-prep high-magnification LA structures are PCME, and for the indirect prep with settling high-mag analyses, 37% of the LA structures are PCME. So, most, but apparently not all, PCME LA fibers are recovered by the indirect prep with settling technique.

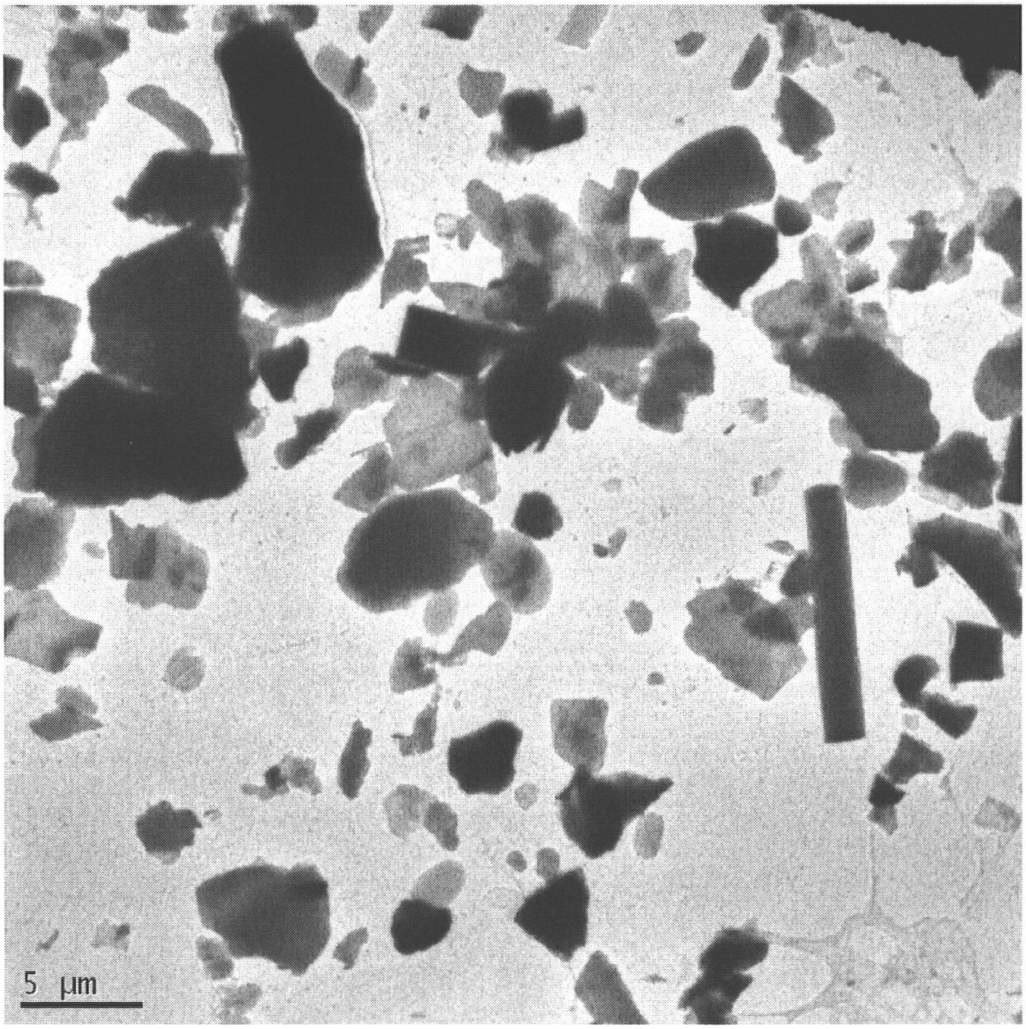

Photo 4: Here is a typical view of one of the spiked rock flour-rich soils prepared for this study after it was ran by FBAS and prepared by indirect-ashed with 3 hours of settling of the aqueous suspension. Most of the coarse rock flour grains, and all of the coarsest ones, have been removed. There are some overlapping particles but in general they are reasonably spread out. The prep is readable. None of the fibers in this photo are LA. The webby material at lower right is undissolved filter medium.

8.0 Summary and Conclusions

ESAT proposes that for the Libby Project, FBAS processing and analysis should proceed by two distinct tracks, depending on the objectives of the study and the quality of data needed. These tracks are:

1) The soil sample should be ran through the FBAS at a soil weight that produces a filter from 10% to 30% loaded that is suitable for direct preparation (i.e., no loose debris or obviously uneven loading). The sample should be prepared for TEM examination by the direct technique whenever possible. If the filter is not properly loaded, it should not be analyzed and the analytical lab should request a new filter submittal. The FBAS facility should then re-run the soil through the FBAS at a lower or higher soil weight in hopes of producing a new filter that is properly loaded.
2) The soil sample should be ran through the FBAS at a soil weight of 5 grams, with the intent of deliberately overloading the filter. The TEM lab should then prepare the sample by indirect-ashed with aqueous suspension and settling for 3 hours as described above. This is the higher-cost approach and may be preferred when the lowest possible detection limit is desired, such as for analysis of background soils.

For the Rock Flour Study, ESAT only ran the tests, compiled the data in an Excel spreadsheet, and wrote this report. The resulting graphs of the data are provided in this report. No attempt was made to apply any statistical treatment to the data. However, it is apparent from a visual examination of the graphs and data that there may be a rough linear correlation between the spiked soil concentration and the TEM-FBA results. The detection limit of the FBAS method as it applies to rock flour-rich soils analyzed indirectly as described in this report may be very low. However, there are not enough data points in this data set to calculate a new detection limit. The purpose of the Rock Flour Study was to develop an analytical approach for optimizing the FBAS method as it applies to soils with very low levels of asbestos and abundant rock flour.

ESAT recommends that a third party should perform a statistical analysis on the data to see how well the TEM-FBA results correlate to the spiked soil concentrations. The 0.1% and 0.05% LA rock flour soils were completely consumed for this study but the remainder of the other 4 are at the Troy SPF in case additional data points are needed.

ESAT recommends that FBAS analysis of BK soils should resume based on the modifications described in this report once the details of the SAP are worked out.

It is not known how Libby soils of other compositions (sandy, organic-rich, etc.) will respond to the new prep technique. The next scheduled FBAS run at the Troy SPF is August 30 to 31, 2011. During this run, 12 soils from the 2008 field season (with EX- sample number prefixes) will be ran by FBAS by both of the tracks described above, for a total of 24 runs. The resulting filter samples will be shipped to the ESAT Region 8 lab for analysis by TEM-ISO.

References

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Webber, James S. et al. Separation and Characterization of Respirable Amphibole Fibers from Libby, Montana (Elutriator Paper). Inhalation Toxicology, volume 20, pages 733-740, 2008.

ESAT Region 8 Rock Flour Study
Rock Flour-Rich Internal Reference Material Soils Analyzed by TEM-FBA
All Analyzed Investigative Samples

TDF: DG244
Aug-11

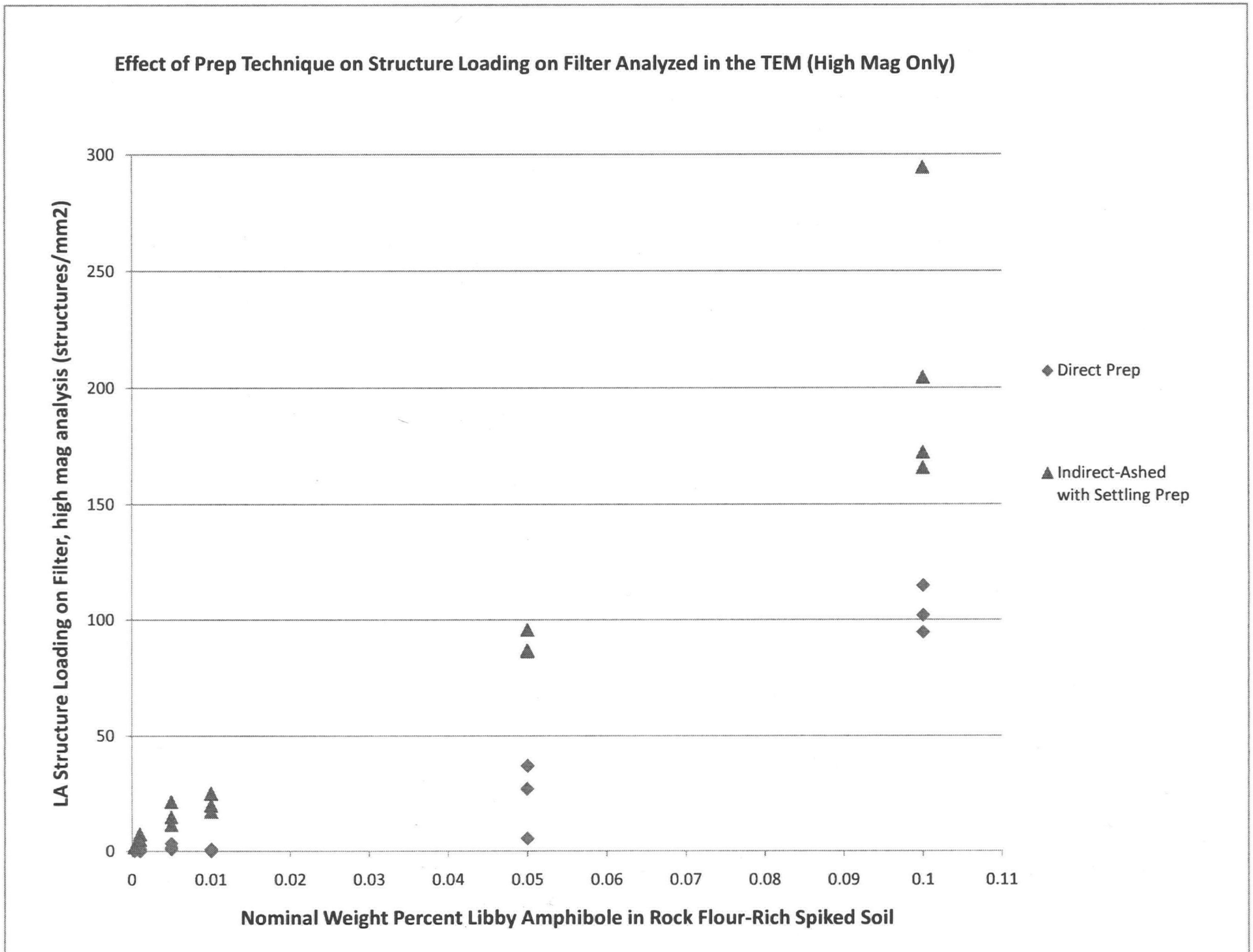
													High Mag Analysis Only					
EPA Sample Number	Nominal Soil LA Content (% weight)	Soil Weight (grams)	Lab	Pre- Treatment	Prep Type	F-factor	Total Suspension Volume (ml)	Volume Applied to Secondary Filter (ml)	Settling Time in Graduated Cylinder	Area Percent Loading by TEM	EFA (mm2)	GO Area (mm2)	Total LA Structure Count	GO's Counted	Area Analyzed (mm2)	LA Structure Filter Loading (s/mm2)	Analytical Sensitivity (s/gram)	LA Soil Concentration (s/gram)
TF-00092	0.1	0.5	ESATR8	None	Direct	1.000	N/A	N/A	N/A	20%	385	0.00943	25	26	0.245	102.0	239,000	5,975,000
TF-00093	0.1	0.5	ESATR8	None	Direct	1.000	N/A	N/A	N/A	20%	385	0.00943	26	24	0.226	114.9	258,000	6,708,000
TF-00094	0.1	0.5	ESATR8	None	Direct	1.000	N/A	N/A	N/A	20%	385	0.00943	25	28	0.264	94.7	223,000	5,575,000
TF-00097	0.1	0.2	ESATR8	Water Suspension	Direct	1.000	N/A	N/A	15 minutes	25%	385	0.00943	25	37	0.349	71.7	472,000	11,800,000
TF-00098	0.1	0.5	ESATR8	Sonication	Direct	1.000	N/A	N/A	N/A	20%	385	0.00943	26	44	0.415	62.7	147,000	3,822,000
TF-00099	0.1	0.5	ESATR8	Sonication	Direct	1.000	N/A	N/A	N/A	20%	385	0.00943	25	22	0.207	120.5	276,000	6,900,000
TF-00102	0.1	0.1	ESATR8	2-Propanol	Direct	1.000	N/A	N/A	15 minutes	15%	385	0.00943	26	49	0.462	56.3	757,000	19,682,000
TF-00103	0.1	5.1	ESATR8	None	Indirect-Ashed	0.200	100	Top 20 ml	30 minutes	30%	346	0.00943	25	16	0.151	165.7	180,000	4,500,000
TF-00104	0.1	5.2	ESATR8	None	Indirect-Ashed	0.250	100	Top 25 ml	1 hour	30%	346	0.00943	25	9	0.085	294.6	252,000	6,300,000
TF-00106	0.1	3.1	ESATR8	None	Indirect-Ashed	0.300	100	Top 30 ml	2 hours	12%	346	0.00943	27	14	0.132	204.5	226,000	6,102,000
TF-00107	0.1	3.6	ESATR8	None	Indirect-Ashed	0.210	100	21 ml of top 50 ml	4 hours	10 to 15%	346	0.00943	26	16	0.151	172.3	240,000	6,240,000
TF-00128	0.05	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	20 to 25%	385	0.0130	26	74	0.962	27.0	64,000	1,664,000
TF-00129	0.05	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	385	0.0130	25	52	0.676	37.0	91,000	2,275,000
TF-00130	0.05	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	385	0.0130	7	98	1.274	5.5	48,000	336,000
TF-00131	0.05	5.0	ESATR8	None	Indirect-Ashed	0.100	100	Top 10 ml	2 hours	15 to 20%	346	0.00978	29	31	0.303	95.7	183,000	5,307,000
TF-00132	0.05	5.0	ESATR8	None	Indirect-Ashed	0.150	100	Top 15 ml	4 hours	15 to 20%	346	0.00978	28	33	0.323	86.8	114,000	3,192,000
TF-00133	0.05	5.0	ESATR8	None	Indirect-Ashed	0.150	100	15 ml of top 50 ml	4 hours	15 to 20%	346	0.00978	27	32	0.313	86.3	118,000	3,186,000
TF-00140	0.05	0.5	ESATR8	Cyclone	Direct	N/A	N/A	N/A	N/A	1%	855	N/A	N/A	N/A	N/A	N/A	N/A	N/A
TF-00141	0.05	1.0	ESATR8	Cyclone	Direct	N/A	N/A	N/A	N/A	1%	855	N/A	N/A	N/A	N/A	N/A	N/A	N/A
TF-00142	0.05	2.0	ESATR8	Cyclone	Direct	N/A	N/A	N/A	N/A	1%	855	N/A	N/A	N/A	N/A	N/A	N/A	N/A
TF-00151	0.05	5.0	ESATR8	Cyclone	Direct	1.000	N/A	N/A	N/A	2%	855	0.00978	0	123	1.203	0.0	1,000	0
TF-00134	0.01	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	20%	385	0.0130	1	95	1.235	0.8	50,000	50,000
TF-00135	0.01	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	20 to 25%	385	0.0130	0	98	1.274	0.0	48,000	0
TF-00136	0.01	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	385	0.0130	1	96	1.248	0.8	49,000	49,000
TF-00137	0.01	4.0	ESATR8	None	Indirect-Ashed	0.150	100	15 ml of top 50 ml	4 hours	10%	346	0.00978	25	102	0.998	25.1	46,000	1,150,000
TF-00138	0.01	4.0	ESATR8	None	Indirect-Ashed	0.200	100	20 ml of top 50 ml	3 hours	15%	346	0.00978	24	123	1.203	20.0	29,000	696,000
TF-00139	0.01	5.0	ESATR8	None	Indirect-Ashed	0.150	100	15 ml of top 50 ml	2 hours	15%	346	0.00978	21	124	1.213	17.3	30,000	630,000
TF-00145	0.005	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	20 to 25%	385	0.0130	1	95	1.235	0.8	50,000	50,000
TF-00146	0.005	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	385	0.0130	2	96	1.248	1.6	49,000	98,000
TF-00165	0.005	0.7	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	385	0.0130	4	94	1.222	3.3	36,000	144,000
TF-00148	0.005	5.0	ESATR8	None	Indirect-Ashed	0.196	102	20 ml of top 50 ml	4 hours	10 to 15%	346	0.00978	18	124	1.213	14.8	23,000	414,000
TF-00149	0.005	5.0	ESATR8	None	Indirect-Ashed	0.147	102	15 ml of top 50 ml	3 hours	15 to 20%	346	0.00978	26	124	1.213	21.4	31,000	806,000
TF-00150	0.005	5.0	ESATR8	None	Indirect-Ashed	0.160	100	16 ml of top 50 ml	2 hours	15%	346	0.00978	14	124	1.213	11.5	29,000	406,000
TF-00153	0.001	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	385	0.0130	0	97	1.261	0.0	49,000	0
TF-00154	0.001	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	385	0.0130	1	96	1.248	0.8	49,000	49,000
TF-00166	0.001	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	385	0.0130	0	95	1.235	0.0	50,000	0
TF-00156	0.001	5.0	ESATR8	None	Indirect-Ashed	0.150	100	15 ml of top 50 ml	2 hours	20 to 25%	346	0.00978	9	124	1.213	7.4	30,000	270,000
TF-00158	0.001	5.0	ESATR8	None	Indirect-Ashed	0.200	100	20 ml of top 50 ml	3 hours	15 to 20%	346	0.00978	6	124	1.213	4.9	23,000	138,000
TF-00159	0.0003	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	385	0.0130	0	96	1.248	0.0	49,000	0
TF-00160	0.0003	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	385	0.0130	0	95	1.235	0.0	50,000	0
TF-00167	0.0003	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	385	0.0130	0	95	1.235	0.0	50,000	0
TF-00162	0.0003	5.0	ESATR8	None	Indirect-Ashed	0.200	100	20 ml of top 50 ml	3 hours	15 to 20%	346	0.00978	2	124	1.213	1.6	23,000	46,000

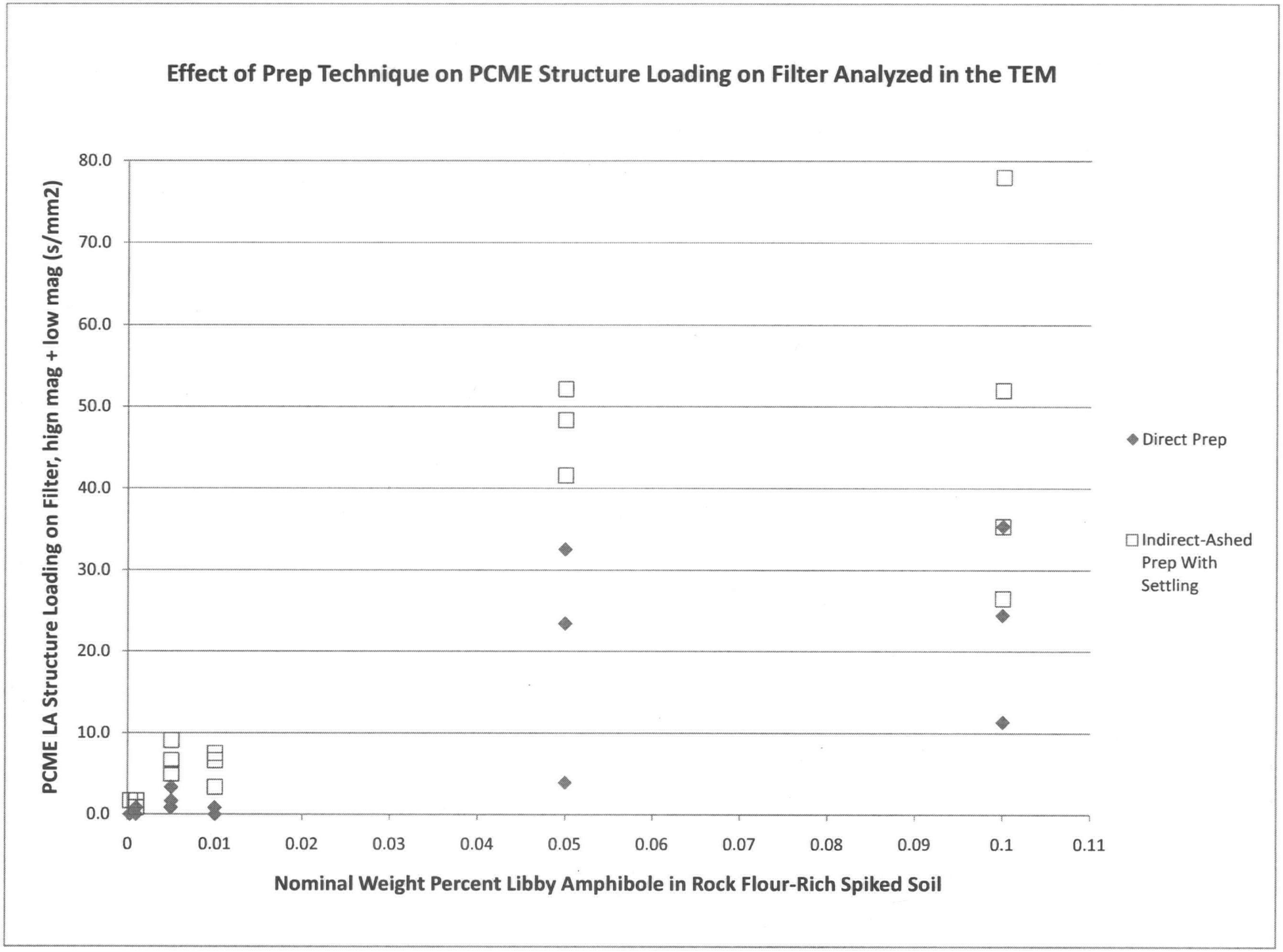
ESAT Region 8 Rock Flour Study

Rock Flour-Rich Internal Reference Material Soils Analyzed by TEM-FBA

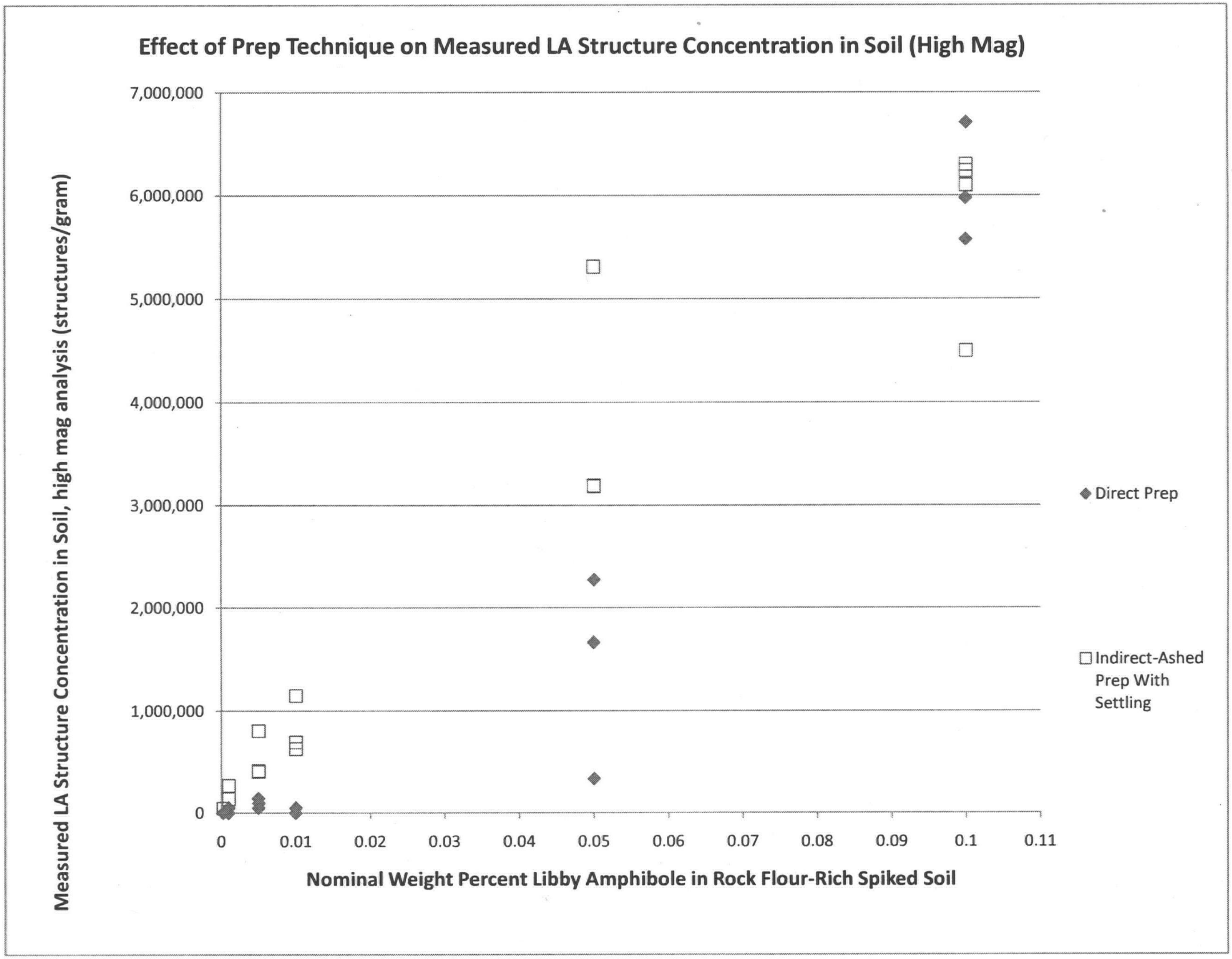
All Analyzed Investigative Samples

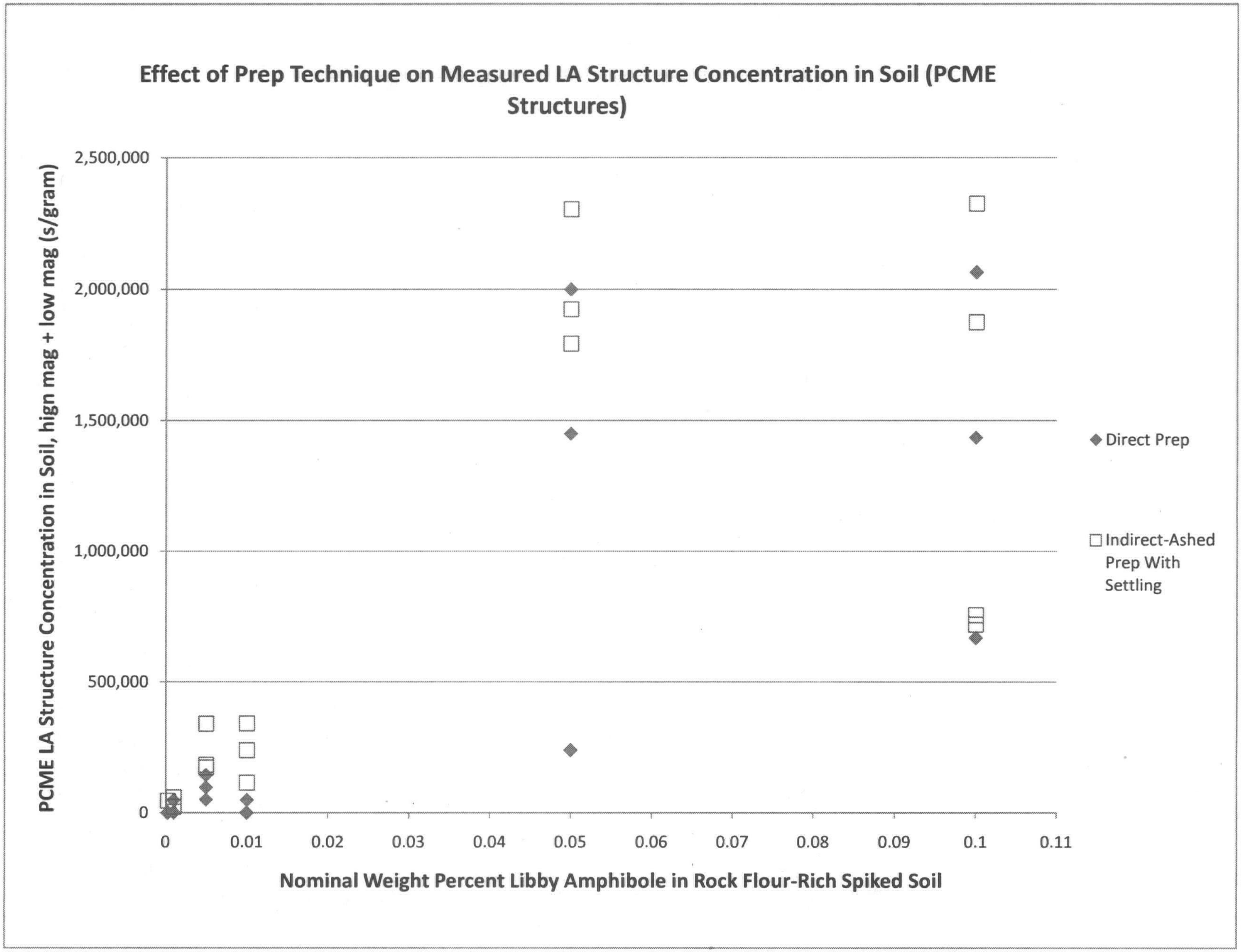
											Analysis for PCME Fibers						
											High Mag	High Mag + Low Mag					
EPA Sample Number	Nominal Soil LA Content (% weight)	Soil Weight (grams)	Lab	Pre- Treatment	Prep Type	F-factor	Total Suspension Volume (ml)	Volume Applied to Secondary Filter (ml)	Settling Time in Graduated Cylinder	Area Percent Loading by TEM	PCME LA Structure Count (high mag only)	PCME LA Structure Count (high mag + low mag)	GO's Counted for PCME Structures (high mag + low mag)	Area Analyzed for PCME Structures (high mag + low mag)	PCME LA Structure Filter Loading (Structures/ mm2)	PCME Analytical Sensitivity (s/gram)	PCME LA Soil Concentration (s/gram)
TF-00092	**0.1**	0.5	ESATR8	None	Direct	1.000	N/A	N/A	N/A	20%	**6**	**6**	**26**	0.245	24.5	239,000	1,434,000
TF-00093	**0.1**	0.5	ESATR8	None	Direct	1.000	N/A	N/A	N/A	20%	**8**	**8**	**24**	0.226	35.3	258,000	2,064,000
TF-00094	**0.1**	0.5	ESATR8	None	Direct	1.000	N/A	N/A	N/A	20%	**3**	**3**	**28**	0.264	11.4	223,000	669,000
TF-00097	**0.1**	0.2	ESATR8	Water Suspension	Direct	1.000	N/A	N/A	15 minutes	25%	**3**	**3**	**37**	0.349	8.6	472,000	1,416,000
TF-00098	**0.1**	0.5	ESATR8	Sonication	Direct	1.000	N/A	N/A	N/A	20%	**11**	**11**	**44**	0.415	26.5	147,000	1,617,000
TF-00099	**0.1**	0.5	ESATR8	Sonication	Direct	1.000	N/A	N/A	N/A	20%	**5**	**5**	**22**	0.207	24.1	276,000	1,380,000
TF-00102	**0.1**	0.1	ESATR8	2-Propanol	Direct	1.000	N/A	N/A	15 minutes	15%	**10**	**10**	**49**	0.462	21.6	757,000	7,570,000
TF-00103	**0.1**	5.1	ESATR8	None	Indirect-Ashed	0.200	100	Top 20 ml	30 minutes	30%	**4**	**4**	**16**	0.151	26.5	180,000	720,000
TF-00104	**0.1**	5.2	ESATR8	None	Indirect-Ashed	0.250	100	Top 25 ml	1 hour	30%	**3**	**3**	**9**	0.085	35.3	252,000	756,000
TF-00106	**0.1**	3.1	ESATR8	None	Indirect-Ashed	0.300	100	Top 30 ml	2 hours	12%	**13**	**25**	**34**	0.321	78.0	93,000	2,325,000
TF-00107	**0.1**	3.6	ESATR8	None	Indirect-Ashed	0.210	100	21 ml of top 50 ml	4 hours	10 to 15%	**8**	**25**	**51**	0.481	52.0	75,000	1,875,000
TF-00128	**0.05**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	20 to 25%	**21**	**25**	**82**	1.066	23.5	58,000	1,450,000
TF-00129	**0.05**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	**21**	**25**	**59**	0.767	32.6	80,000	2,000,000
TF-00130	**0.05**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	**5**	**5**	**98**	1.274	3.9	48,000	240,000
TF-00131	**0.05**	5.0	ESATR8	None	Indirect-Ashed	0.100	100	Top 10 ml	2 hours	15 to 20%	**12**	**24**	**59**	0.577	41.6	96,000	2,304,000
TF-00132	**0.05**	5.0	ESATR8	None	Indirect-Ashed	0.150	100	Top 15 ml	4 hours	15 to 20%	**17**	**26**	**51**	0.499	52.1	74,000	1,924,000
TF-00133	**0.05**	5.0	ESATR8	None	Indirect-Ashed	0.150	100	15 ml of top 50 ml	4 hours	15 to 20%	**14**	**26**	**55**	0.538	48.3	69,000	1,794,000
TF-00140	**0.05**	0.5	ESATR8	Cyclone	Direct	N/A	N/A	N/A	N/A	1%	N/A	N/A	N/A	N/A	N/A	N/A	N/A
TF-00141	**0.05**	1.0	ESATR8	Cyclone	Direct	N/A	N/A	N/A	N/A	1%	N/A	N/A	N/A	N/A	N/A	N/A	N/A
TF-00142	**0.05**	2.0	ESATR8	Cyclone	Direct	N/A	N/A	N/A	N/A	1%	N/A	N/A	N/A	N/A	N/A	N/A	N/A
TF-00151	**0.05**	5.0	ESATR8	Cyclone	Direct	1.000	N/A	N/A	N/A	2%	**0**	**0**	**123**	1.203	0.0	1,000	0
TF-00134	**0.01**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	20%	**1**	**1**	**95**	1.235	0.8	50,000	50,000
TF-00135	**0.01**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	20 to 25%	**0**	**0**	**98**	1.274	0.0	48,000	0
TF-00136	**0.01**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	**1**	**1**	**96**	1.248	0.8	49,000	49,000
TF-00137	**0.01**	4.0	ESATR8	None	Indirect-Ashed	0.150	100	15 ml of top 50 ml	4 hours	10%	**9**	**9**	**123**	1.203	7.5	38,000	342,000
TF-00138	**0.01**	4.0	ESATR8	None	Indirect-Ashed	0.200	100	20 ml of top 50 ml	3 hours	15%	**4**	**4**	**123**	1.203	3.3	29,000	116,000
TF-00139	**0.01**	5.0	ESATR8	None	Indirect-Ashed	0.150	100	15 ml of top 50 ml	2 hours	15%	**8**	**8**	**124**	1.213	6.6	30,000	240,000
TF-00145	**0.005**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	20 to 25%	**1**	**1**	**95**	1.235	0.8	50,000	50,000
TF-00146	**0.005**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	**2**	**2**	**96**	1.248	1.6	49,000	98,000
TF-00165	**0.005**	0.7	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	**4**	**4**	**94**	1.222	3.3	36,000	144,000
TF-00148	**0.005**	5.0	ESATR8	None	Indirect-Ashed	0.196	102	20 ml of top 50 ml	4 hours	10 to 15%	**8**	**8**	**124**	1.213	6.6	23,000	184,000
TF-00149	**0.005**	5.0	ESATR8	None	Indirect-Ashed	0.147	102	15 ml of top 50 ml	3 hours	15 to 20%	**11**	**11**	**124**	1.213	9.1	31,000	341,000
TF-00150	**0.005**	5.0	ESATR8	None	Indirect-Ashed	0.160	100	16 ml of top 50 ml	2 hours	15%	**6**	**6**	**124**	1.213	4.9	29,000	174,000
TF-00153	**0.001**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	**0**	**0**	**97**	1.261	0.0	49,000	0
TF-00154	**0.001**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	**1**	**1**	**96**	1.248	0.8	49,000	49,000
TF-00166	**0.001**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	**0**	**0**	**95**	1.235	0.0	50,000	0
TF-00156	**0.001**	5.0	ESATR8	None	Indirect-Ashed	0.150	100	15 ml of top 50 ml	2 hours	20 to 25%	**2**	**2**	**124**	1.213	1.6	30,000	60,000
TF-00158	**0.001**	5.0	ESATR8	None	Indirect-Ashed	0.200	100	20 ml of top 50 ml	3 hours	15 to 20%	**1**	**1**	**124**	1.213	0.8	23,000	23,000
TF-00159	**0.0003**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	**0**	**0**	**96**	1.248	0.0	49,000	0
TF-00160	**0.0003**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	15 to 20%	**0**	**0**	**95**	1.235	0.0	50,000	0
TF-00167	**0.0003**	0.5	EMSL27	None	Direct	1.000	N/A	N/A	N/A	10 to 15%	**0**	**0**	**95**	1.235	0.0	50,000	0
TF-00162	**0.0003**	5.0	ESATR8	None	Indirect-Ashed	0.200	100	20 ml of top 50 ml	3 hours	15 to 20%	**2**	**2**	**124**	1.213	1.6	23,000	46,000


Effect of Prep Technique on Structure Loading on Filter Analyzed in the TEM (High Mag Only)
LA Structure Loading on Filter, high mag analysis (structures/mm2)
300
250
200
150
100
50
0
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
Nominal Weight Percent Libby Amphibole in Rock Flour-Rich Spiked Soil
Direct Prep
Indirect-Ashed with Settling Prep



Effect of Prep Technique on PCME Structure Loading on Filter Analyzed in the TEM
PCME LA Structure Loading on Filter, hign mag + low mag (s/mm2)
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
Nominal Weight Percent Libby Amphibole in Rock Flour-Rich Spiked Soil
Direct Prep
Indirect-Ashed Prep With Settling


Effect of Prep Technique on Measured LA Structure Concentration in Soil (High Mag)
Measured LA Structure Concentration in Soil, high mag analysis (structures/gram)
7,000,000
6,000,000
5,000,000
4,000,000
3,000,000
2,000,000
1,000,000
0
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
Nominal Weight Percent Libby Amphibole in Rock Flour-Rich Spiked Soil
Direct Prep
Indirect-Ashed Prep With Settling



Effect of Prep Technique on Measured LA Structure Concentration in Soil (PCME Structures)
PCME LA Structure Concentration in Soil, hign mag + low mag (s/gram)
2,500,000
2,000,000
1,500,000
1,000,000
500,000
0
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
Nominal Weight Percent Libby Amphibole in Rock Flour-Rich Spiked Soil
Direct Prep
Indirect-Ashed Prep With Settling